
**Nickel alloys — Determination
of tantalum — Inductively
coupled plasma optical emission
spectrometric method**

*Alliages de nickel — Détermination du tantale — Méthode par
spectrométrie d'émission optique avec source à plasma induit par
haute fréquence*

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 155, *Nickel and nickel alloys*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Nickel alloys — Determination of tantalum — Inductively coupled plasma optical emission spectrometric method

1 Scope

This document specifies an inductively coupled plasma optical emission spectrometric method for the determination of tantalum contents between 0,1 % and 5 % in nickel alloys.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 648, *Laboratory glassware — Single-volume pipettes*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

4 Principle

Dissolution of a test portion in a mixture of hydrofluoric, hydrochloric, nitric and phosphoric acid and fuming after addition of perchloric acid. Addition of hydrofluoric acid and, if desired, of an internal reference element and dilution of the solution to known volume. Nebulization of the solution into an inductively coupled plasma optical emission spectrometer and measurement of the intensity of the emitted light from tantalum, and, where appropriate, from the internal reference element, simultaneously.

An example of the wavelength for tantalum is given in [Table 1](#).

The method uses a calibration based on a very close matrix-matching of the calibration solutions to the sample and bracketing between 0,75 and 1,25 of the approximate content of tantalum in the sample to be analysed. The content of all elements in the sample has, therefore, to be approximately known. If the contents are not known, the sample has to be analysed by some semi-quantitative method. The advantage of this procedure is that all possible interferences from the matrix will be compensated, which will result in high accuracy. This is most important for spectral interferences, which can be severe in very highly alloyed matrixes. All possible interferences shall be kept at a minimum level. Therefore, it is essential that the spectrometer used meets the performance criteria specified in the method for the selected wavelengths.

The line corresponding to 240,06 nm has been investigated. If other lines are used, they shall be carefully checked. The wavelength for the internal reference element should be selected carefully. The

use of scandium at 363,07 nm is recommended. This wavelength is interference-free for the elements and contents generally found in nickel alloys.

Table 1 — Example of wavelength for tantalum

Element	Wavelength	Interferences
Tantalum	240,06	Co - Fe - Hf

NOTE The use of an internal reference element is not essential since no relevant differences between laboratories operating with or without internal reference elements were found when the precision test was carried out (see 9.2.2).

5 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only grade 2 water as specified in ISO 3696.

5.1 Hydrofluoric acid, HF, $\rho = 1,14$ g/ml, or $\rho = 1,17$ g/ml.

WARNING — Hydrofluoric acid is extremely irritating and corrosive to skin and mucous membranes producing severe skin burns which are slow to heal. In the case of contact with skin, wash well with water, apply a topical gel containing 2,5 % (mass fraction) calcium gluconate, and seek immediate medical treatment.

5.2 Hydrochloric acid, HCl, $\rho = 1,19$ g/ml.

5.3 Nitric acid, HNO₃, $\rho = 1,40$ g/ml.

5.4 Phosphoric acid, H₃PO₄, $\rho = 1,70$ g/ml.

5.5 Perchloric acid, HClO₄, $\rho = 1,54$ g/ml, or $\rho = 1,67$ g/ml.

WARNING — Perchloric acid vapour may cause explosions in the presence of ammonia, nitrous fumes or organic material in general. The use of fume hoods (water scrubbed) when using perchloric acid is highly recommended.

5.6 Internal reference element solution, 100 mg/l.

Choose a suitable element to be added as internal reference and prepare a 100 mg/l solution.

5.7 Tantalum standard solution, 10 g/l.

Weigh, to the nearest 0,001 g, 1 g of tantalum [minimum 99,9 % (mass fraction)], place it in a beaker (6.1), cover with an appropriate lid and dissolve it in a mixture of 10 ml of hydrofluoric acid (5.1) and 10 ml of nitric acid (5.3).

Cool and transfer quantitatively into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 10 mg of tantalum.

5.8 Tantalum standard solution, 1 g/l.

Weigh, to the nearest 0,000 5 g, 0,1 g of tantalum [minimum 99,9 % (mass fraction)], place it in a beaker (6.1), cover with an appropriate lid and dissolve it in a mixture of 10 ml of hydrofluoric acid (5.1) and 10 ml of nitric acid (5.3).

Cool and transfer quantitatively into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 1 mg of tantalum.

5.9 Tantalum standard solution, 100 mg/l.

Transfer 10 ml of the tantalum standard solution (5.8) into a 100 ml one-mark volumetric flask. Add 10 ml of hydrofluoric acid (5.1) and 10 ml of nitric acid (5.3). Dilute to the mark with water and mix.

1 ml of this solution contains 0,1 mg of tantalum.

5.10 Standard solutions of matrix elements.

Prepare standard solutions for each element for which the content is higher than 1 % in the test sample. Use pure metals or chemical substances with contents of tantalum less than 10 µg/g.

6 Apparatus

All volumetric glassware shall be class A and calibrated in accordance with ISO 648 or ISO 1042, as appropriate.

Ordinary laboratory apparatus and the following.

6.1 Polytetrafluoroethylene (PTFE) beakers or perfluoroalkoxy-polymer (PFA) beakers, preferably with a graphite base.

6.2 Polypropylene volumetric flasks, of capacity 100 ml, calibrated in accordance with ISO 1042.

6.3 Optical emission spectrometer, equipped with inductively coupled plasma.

This shall be equipped with a nebulization system resistant to hydrofluoric acid. The instrument used will be satisfactory if, after optimising in accordance with 8.3, it meets the performance criteria given in this subclause.

The spectrometer can be either a simultaneous or a sequential one. If a sequential spectrometer can be equipped with an extra arrangement for simultaneous measurement of the internal reference element wavelength, it can be used with the internal reference method. If the sequential spectrometer is not equipped with this arrangement, an internal reference cannot be used and an alternative method without an internal reference element should be applied.

Calculate the bandwidth (full width at half maximum), in accordance with A.2, for the line used and for the line of the internal reference element. The bandwidth shall be less than 0,030 nm.

Calculate the standard deviation of 10 measurements of the absolute intensity or of the intensity ratio between tantalum and the internal reference element, by using the most concentrated calibration solution for tantalum in accordance with A.3. The relative standard deviation shall not exceed 0,4 %.

Calculate the background equivalent concentration (BEC) in accordance with A.4, for the analytical line using a solution containing only the analyte element. The maximum values of BEC obtained should be 0,8 mg/l.

7 Sampling and sample preparation

Sampling and preparation of the laboratory sample shall be carried out by normal agreed procedures or, in case of dispute, by appropriate national standards.

The laboratory sample is normally in the form of millings or drillings. No further preparation is necessary.

If it is suspected that the laboratory sample is contaminated with oil or grease from the milling or drilling process, it shall be cleaned with high purity acetone and then dried in air.

If brazed alloy tools are used in the preparation of the laboratory sample, the sample shall be further cleaned by pickling in 15 % (mass fraction) nitric acid for a few minutes. It shall then be rinsed several times with distilled water, followed by washing in acetone and drying in air.

8 Procedure

8.1 Test portion

Weigh, to the nearest 0,001 g, 0,25 g of the test sample.

8.2 Preparation of test solution, T_{TA}

A PTFE or PFA beaker (6.1) shall be used when using HF (5.1).

8.2.1 Place the test portion in a PTFE or PFA beaker, preferably with a graphite base.

8.2.2 Add 5 ml of hydrofluoric acid (5.1), 30 ml of hydrochloric acid (5.2), 3 ml of nitric acid (5.3), cover with an appropriate lid and allow the dissolution of the sample to take place overnight at room temperature. Then, add 2,5 ml of phosphoric acid (5.4). If necessary, heat to complete dissolution. Add 7,5 ml of perchloric acid (5.5) and heat until the perchloric acid starts to fume. Continue to fume for 2 min to 3 min.

NOTE 5 ml of phosphoric acid (5.4) and 5 ml of perchloric acid (5.5) can also be used, instead of 2,5 ml of phosphoric acid (5.4) and 7,5 ml of perchloric acid (5.5).

8.2.3 Cool the solution and add 10 ml of water to dissolve the salts. Some residues may remain undissolved. In this case, add 2 ml of hydrofluoric acid (5.1) and heat gently for about 20 min until the residues dissolve completely.

8.2.4 The following is an alternative dissolution procedure for 8.2.2 and 8.2.3.

Add 30 ml of hydrochloric acid (5.2), 3 ml of nitric acid (5.3) and 5 ml of phosphoric acid (5.4), or 20 ml of hydrochloric acid (5.2), 10 ml of nitric acid (5.3) and 5 ml of phosphoric acid (5.4). Cover with an appropriate lid and let the dissolution begin at room temperature. If necessary, heat to complete dissolution. Add 2 ml of hydrofluoric acid (5.1) and 5 ml of sulphuric acid ($\rho = 1,84$ g/ml) and heat until sulfuric acid starts to fume. Cool the solution and add 10 ml of water to dissolve the salts. Heat gently until the residues dissolve completely.

8.2.5 Cool the solution to room temperature and transfer the solution quantitatively to a 100 ml volumetric polypropylene flask. If an internal reference element solution (5.6) is used, add 10 ml of this solution.

8.2.6 Dilute to the mark with water and mix. Proceed as quickly as possible to the measurement.

8.3 Optimization of the spectrometer

8.3.1 Start the inductively coupled plasma optical emission spectrometer and let it stabilize in accordance with the manufacturer's instructions before taking any measurements.

8.3.2 Optimize the instrument in accordance with the manufacturer's instructions.

8.3.3 Prepare the software for measurements of the intensity, and for the calculation of the mean value and relative standard deviation corresponding to the appropriate wavelengths.

8.3.4 If an internal reference element is used, prepare the software to calculate the ratio between the intensity of the analyte and that of the internal reference element. The intensity of the internal reference element shall be measured simultaneously with that of the analyte.

8.3.5 Check the instrument performance criteria given in [6.3](#).

8.4 Pre-determination of the test solution

8.4.1 Prepare a calibration solution, K_x, matrix matched to the test sample solution as follows.

8.4.2 In a 100 ml volumetric polypropylene flask ([6.2](#)) marked K_x, prepare a calibration solution K_x, corresponding to the approximate content of tantalum in the sample, in per cent, as indicated in [Table 2](#).

Table 2 — Pre-determination of the test solution

Estimated content %	K _x equivalent content %	K _x identification	Tantalum standard solution	Standard solution volume ml
0,10 to 1,0	1,0	K1	5.9	2,5
1,0 to 5,0	5,0	K5	5.9	12,5

8.4.3 In this volumetric flask K_x, add volumes of standard solutions ([5.10](#)) necessary to match the sample matrix to be tested, for each element for which the content is above 1 %.

The matrix should be matched to the nearest per cent.

8.4.4 Add 2,5 ml of phosphoric acid ([5.4](#)) and 7,5 ml of perchloric acid ([5.5](#)) and 10 ml of the internal reference element solution ([5.6](#)). Dilute to the mark with water and mix.

8.4.5 Also prepare a zero member solution, K₀, prepared in the same way as the calibration solution K_x omitting tantalum.

8.4.6 Measure the absolute intensities (I_0 and I_x) for the solutions K₀ and K_x.

8.4.7 Measure the absolute intensity I_{TTa} for the test solution T_{Ta}.

8.4.8 Calculate the approximate content of tantalum, w_{TTa} , in percent, in the test solution, by means of [Formula \(1\)](#):

$$w_{TTa} = \frac{I_{TTa} (w_x - w_0)}{I_x - I_0} \quad (1)$$

8.5 Preparation of calibration solutions for bracketing, K_{l,Ta} and K_{h,Ta}

8.5.1 For each test solution T_{Ta} prepare two matrix matched calibration solutions, K_{l,Ta} and K_{h,Ta}, with the tantalum concentrations in K_{l,Ta} slightly below, and in K_{h,Ta} slightly above, the concentration in the unknown test solution as described in [8.5.2](#) to [8.5.5](#).